

TECHNICAL MEMORANDUM MAN-64-4

THE SYNTHESIS AND EVALUATION OF MERCURIAL (II) ACETATES  
AND RELATED TRIPHENYL PHOSPHINE COMPLEXES AS HIGH-  
TEMPERATURE LUBRICANT ADDITIVES

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13. ABSTRACT (Maximum 200 words) The purpose of this study was to synthesize and evaluate the following compounds as potential additives to lubricants applied to aircraft systems: Mercury (II) acetate derivatives, triphenylphosphine complexes of Mercury (II) acetate derivatives, and Mercury (II) acetamide.				
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### FOREWORD

This first semi-annual report on the internal program, "Synthesis and Evaluation of Mercurial (II) Acetates and Related Triphenyl-Phosphine Complexes as Lubricant Additives," was initiated under Project No. 3044, Task No. 304405, with Captain Stanley M. Dec acting as project engineer.

This report covers work conducted from 1 May 1963 to 31 October 1963.

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TM-MAN-64-4

The synthesis and evaluation of mercury (II) acetate derivatives and related triphenylphosphine complexes as lubricant additives.

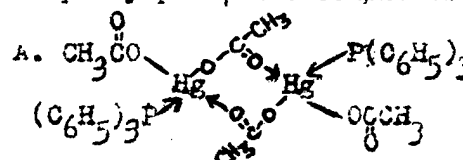
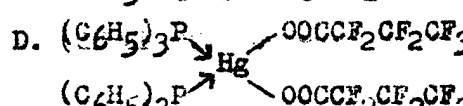
## 1. Introduction

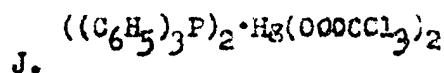
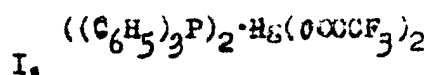
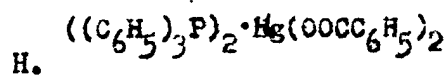
The purpose of this study was to synthesize and evaluate the following compounds as potential additives to new lubricants applied to aircraft systems:

### 1. Mercury (II) Acetate Derivatives.

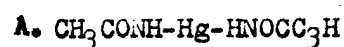
- A.  $\text{CH}_3\text{COO}-\text{Hg}-\text{OOCCH}_3$
- B.  $\text{CH}_3\text{CH}_2\text{COO}-\text{Hg}-\text{OOCCH}_2\text{CH}_3$
- C.  $\text{CH}_3\text{CH}_2\text{CH}_2\text{COO}-\text{Hg}-\text{OOCCH}_2\text{CH}_2\text{CH}_3$
- D.  $\text{CF}_3\text{CF}_2\text{CF}_2\text{COO}-\text{Hg}-\text{OOCCF}_2\text{CF}_2\text{CF}_3$
- E.  $\text{HOCH}_2\text{COO}-\text{Hg}-\text{OOCCH}_2\text{OH}$
- F.  $\text{ClCH}_2\text{COO}-\text{Hg}-\text{OOCCH}_2\text{Cl}$
- G.  $\text{BrCH}_2\text{COO}-\text{Hg}-\text{OOCCH}_2\text{Br}$
- H.  $\text{C}_6\text{H}_5\text{COO}-\text{Hg}-\text{OOCCH}_2\text{C}_6\text{H}_5$
- I.  $\text{CF}_3\text{COO}-\text{Hg}-\text{OOCCH}_2\text{CF}_3$
- J.  $\text{CCl}_3\text{COO}-\text{Hg}-\text{OOCCH}_2\text{CCl}_3$

### 2. Triphenylphosphine Complexes of Mercury (II) Acetate Derivatives

- A. 
- B.  $((\text{CH}_3\text{CH}_2\text{COO})_2\text{Hg})_2 \cdot ((\text{P}(\text{C}_6\text{H}_5)_3)_2)$  (bridge structure 2A)
- C.  $((\text{CH}_3\text{CH}_2\text{CH}_2\text{COO})_2\text{Hg})_2 \cdot ((\text{P}(\text{C}_6\text{H}_5)_3)_2)$  (bridge structure 2A)
- D. 
- E.  $((\text{C}_6\text{H}_5)_3\text{P})_2 \cdot \text{Hg}(\text{OOCCH}_2\text{OH})_2$
- F.  $((\text{C}_6\text{H}_5)_3\text{P})_2 \cdot \text{Hg}(\text{OOCCH}_2\text{Cl})_2$
- G.  $((\text{C}_6\text{H}_5)_3\text{P})_2 \cdot \text{Hg}(\text{OOCCH}_2\text{Br})_2$



### 3. Mercury (II) Acetamide



The results are summarized in Tables I and II.

This study was approached through a survey of the literature in specific areas and through experimental investigations of the above compounds.

## II. Preparation of Compounds

### A. Synthesis of Mercury (II) Acetates

It was found that the mercurial (II) acetates could readily be prepared by the reaction of the organic acid with yellow mercuric oxide:



The general procedure involved short periods of refluxing to promote the reaction;

The reaction proceeded at room temperature with perfluorobutyric acid.

1. Mercury (II) acetate was a commercial preparation obtained from Matheson, Coleman, and Bell.

2. Synthesis of Mercury (II) propionate. To a solution of 74.04 grams (1 mole) of propionic acid in 150cc. of reagent grade benzene was added 102 grams (0.472 moles) of yellow mercuric oxide. The mercuric oxide was completely reacted after 30 minutes of refluxing. The colorless solution was filtered hot and allowed

to cool to room temperature. The crystallized, colorless plates were collected on a suction filter and gave 100 grams (76.5%) of product, *m.p.*, 111.5-112.5°.

3. Synthesis of mercury (II) butyrate. To a solution of 88 grams (1 mole) of butyric acid in 200 cc. of reagent grade benzene was added 120 grams (0.555 moles) of yellow mercuric oxide. The reaction mixture was refluxed for 20 minutes. During this time all of the mercuric oxide was reacted. An excess of 20 grams of yellow mercuric oxide was then added and the mixture refluxed for an additional 30 minutes. The mixture was filtered hot to remove the excess mercuric oxide. The filtrate was cooled to room temperature and the precipitated solid was collected on a suction filter, yielding 110 grams (29.1%) of white plates, *m.p.* 95-96°.

4. Synthesis of mercury (II) chloroacetate. To a solution of 40.83 grams (0.432 moles) of chloroacetic acid dissolved in 250 cc. of reagent grade benzene was added 46.8 grams (0.216 moles) of yellow mercuric oxide. The reaction was allowed to reflux for 21 hours. The hot solution was then filtered to remove traces of unreacted mercuric oxide. The hot filtrate was treated with charcoal, refiltered, and cooled. The precipitated white solid was collected on a suction filter in 63 grams (37.7%) yield, *m.p.* 114-115°.

5. Synthesis of Mercury (II) Perfluorobutyrate. To a solution of 100 grams (0.467 moles) of perfluorobutyric acid in 250 cc. of reagent grade benzene was added 45.3 grams (0.209 moles) of yellow mercuric oxide. The reaction proceeded at room temperature, overnight, for a period of 17 hours. The reaction mixture was treated with activated carbon and filtered. The solvent and unreacted butyric acid were evaporated under vacuum and gave 90 grams (30.7%) of a pale brown liquid.

6. Synthesis of Mercury (II) Benzoate. To 700 cc. of reagent grade benzene was added 40 grams (0.10 moles) of benzoic acid and 10.8 grams (0.05 moles) of yellow mercuric oxide. The reaction mixture was refluxed for two hours, filtered hot, and cooled to room temperature. The precipitated white needles were collected on a suction filter in a 41 gram (93.6%) yield, *m.p.* 110-112°.

7. Synthesis of Mercury (II) Trifluoroacetate. To a stirred suspension of 47.2 grams (0.218 moles) of yellow mercuric oxide in 100 cc. of reagent grade benzene was slowly added 100 grams (0.435 moles) of trifluoroacetic acid dissolved in 100 cc. of benzene. Additional mercuric oxide was added until a slight excess of unreacted mercuric oxide remained. The reaction mixture was heated and treated with activated carbon. After cooling, the white crystalline solid was collected on a suction funnel and gave a 175 gram (94.3%) yield, *m.p.* 122-125°.

8. Synthesis of Mercury (II) Trichloroacetate. To a solution of 80.5 grams (0.303 moles) of trichloroacetic acid in 250 cc. of reagent grade benzene was added 30 grams (0.138 moles) of yellow mercuric oxide. The reaction mixture was refluxed for 4 hours. At the end of this time period, the mercuric oxide was completely reacted. The reacted solution was cooled to 15° and the precipitated white crystalline solid was collected on a suction filter in a 50 gram (68.8%) yield, *m.p.* 127 dec.

9. Synthesis of Mercury (II) Hydroxyacetate. To a solution of 100 grams (1.32 moles) of hydroxyacetic acid in 250 cc. of reagent grade benzene was added 128 grams (0.595 moles) of yellow mercuric oxide. The reaction proceeded at room temperature to give a white solid. Refluxing was continued for an additional hour. After cooling, the white solid was collected on a suction filter, crystallized from hot  $\text{CCl}_4$  and gave 85 grams (40.8%) of product, *m.p.* 114-115.

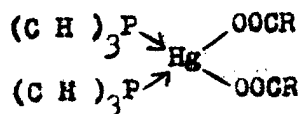


10. Synthesis of Mercury (II) Acetamide. To an Erlenmeyer flask was added 118 grams (2 moles) of acetamide and 108,3 grams (0.5 mole) of yellow mercuric oxide. The mixture was gradually heated above 180°C on a hot plate until all of the mercuric oxide was reacted. After cooling, the white solid was crystallized from hot methanol and gave 110 grams (70%) of white crystalline solid, *m.p.* 195°C.

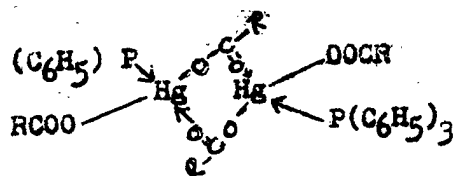
B. Synthesis of Triphenylphosphine Complexes of Mercury (II) Acetates.

The synthesis of compounds of triphenylphosphine with mercury (II) acetates was undertaken to develop additives of higher molecular weight. The derivatives synthesized were found to give two classes of compounds:

A 4-covalent complex with tetrahedral structure,



and a 4-covalent trans-symmetric tetragonal bridged structure:



These structural assignments were assigned on the basis of work performed by Mann<sup>1,2</sup> et al. on the "Addition Compounds" derived from 3-covalent phosphine with Cd and Hg<sup>II</sup> halides.

The compounds were conveniently prepared by a reaction of equal molar quantities of triphenylphosphine with the acetate in dry solvent (ether, tetrahydrofuran, or 1,4-dioxane).

1. Synthesis of Diacetato bistrisphenylphosphine-w-diacetatodimercury

To a solution of 33.24 grams (0.1042 moles) of mercury (II) acetate in 250 cc. of absolute ether was slowly added 20.43 grams (0.078 moles) of triphenylphosphine. The reaction mixture was shaken and cooled (to prevent product decomposition) during the addition. The reaction mixture was then stirred for 15 minutes. The precipitated white crystals were collected on a suction filter and rinsed twice with 40 cc. portions of dry tetrahydrofuran. The yield of product obtained was 50 grams (82.5%), m.p., dec.

ANAL: Calcd. For  $C_{22}H_{21}HgO_4P$ :

%C 45.51 %H, 3.13 %O, 11.02 %P 5.16

FOUND: %C 45.62 %H, 3.91 %O, 11.14 %P 4.57

2. Synthesis of Dipropionatobistriphenylphosphine-w-dipropionatedimercury.

To a solution of 5 grams (0.014 moles) of mercury (II) propionate in 40 cc of dry tetrahydrofuran (distilled from  $LiAlH_4$ ) was slowly added 3.8 grams (0.0145 moles) of triphenylphosphine. The reaction mixture was stirred and cooled during the addition to prevent product decomposition. The precipitated white crystals were collected on a suction filter, rinsed with 25 cc of absolute ether and gave 8 grams (47.0% of white solid, m.p. 263-264.5°.

ANAL; Calcd for:  $C_{48}H_{50}O_8P_2Hg_2$

%C 47.31 %H 4.14 %P 5.08

FOUND: %C 50.82 %H 4.52 %P 6.60

3. Synthesis of Dibutyratobistriphenylphosphine-w-dibutyratodimercury.

To a solution of 5 grams (0.013 moles) of mercury (II) butyrate dissolved in 40 cc of dry tetrahydrofuran (distilled from  $LiAlH_4$ ) was slowly added, 3.5 grams (0.013 moles) of triphenylphosphine. The reaction mixture was stirred and cooled to prevent product decomposition. The precipitated white solid was collected on a suction filter and gave 7 grams (42.3%) of product, m.p., 151-152°.

ANAL: Calcd for:  $C_{52}H_{60}O_8P_2Hg_2$

%C 48.99 %H 4.58

Found: %C 49.21 %H 4.96

4. Synthesis of Diperfluoroacetatobistriphenylphosphine-mercury. To a

solution of 10 grams (0.016 moles) of mercury (II) perfluorobutyrate was slowly added, 4.18 grams (0.016 moles) of triphenylphosphine, while the temperature of the reaction flask was kept cooled to room temperature. A white solid settled out immediately. The reaction mixture was stirred and cooled to prevent product decomposition. The white crystalline solid was collected in 11 gram (63.2%) yield, mp., 220-1°.

ANAL: Calcd for:  $C_{44}H_{30}F_{14}O_4P_2$   
%C 45.01 %H 2.54 %P 5.38  
Found: %C 45.60 %H 2.83 %P 6.44

5. Synthesis of Dihydroxyacetobistriphenylphosphinemercury. To a suspension of 5 grams (0.016 moles) of mercury (II) hydroxyacetate was slowly added 4.35 grams (0.016 moles) of triphenylphosphine. The reaction mixture was stirred, and cooled to prevent product decomposition. The white crystals were collected on a suction filter in a 8.4 gram (71.8%) yield, m.p., 195°.

ANAL: Calcd for:  $C_{40}H_{34}O_6P_2$   
%C 54.99 %H 3.91 %P 7.10  
Found: %C 54.08 %H 4.23 %P 6.78

6. Synthesis of Dichloracetobistriphenylphosphinemercury. To a solution of 12.01 grams (0.0124 moles) of mercury (II) chloroacetate in 200 cc of dry tetrahydrofuran (distilled from  $LiAlH_4$ ) was slowly added 13.62 grams (0.0518 moles) of triphenylphosphine. The reaction mixture was stirred and cooled to prevent product decomposition. The precipitated solid was collected on a suction filter in a 11.75 gram (58.2%) yield, m.p, 192° dec.

ANAL: Calcd for:  $C_{40}H_{34}P_2H_2O_4Cl_2$   
%C 52.61 %H 3.76 %Cl 7.79 %P 6.80  
Found: %C 50.82 %H 3.64 %Cl 8.21 %P 5.85

7. Synthesis of Dibenzoatobistriphenylphosphinemercury. To a solution of 10 grams (0.0226 moles) of mercury (II) benzoate in 50 cc of dry tetrahydrofuran (distilled from  $\text{LiAlH}_4$ ) was slowly added 5.92 grams (0.0226 moles) of triphenylphosphine. The reaction mixture was stirred and cooled to prevent product decomposition. The solvent was evaporated and yielded 13.9 grams (64.1%) of white crystals m.p., 204-5.

ANAL: Calcd for:  $\text{C}_{50}\text{H}_{40}\text{P}_2\text{HgO}_4$   
%C 62.05 %H 4.16 %P 6.41 %Hg 20.64  
FOUND: %C 62.40 %H 4.64 %P 6.37 %Hg 21.49

8. Synthesis of Ditrifluoroacetobistriphenylphosphinemercury. To a solution of 11.08 grams (0.025 moles) of mercury (II) trifluoroacetate in 100cc of dry 1,4-dioxane (distilled from  $\text{LiAlH}_4$ ) was slowly added 6.81 grams (0.026 moles) of triphenylphosphine. The reaction was stirred and cooled to prevent product decomposition. The white solid was collected on a suction filter in a 10 gram (61.6%) yield, m.p., 230-1°.

ANAL: Calcd for:  $\text{C}_{40}\text{H}_{30}\text{F}_6\text{O}_4\text{P}_2$   
%C 50.51 %H 3.16 %P 6.52  
FOUND: %C 50.58 %H 3.28 %P 7.57

9. Synthesis of Ditrichloroacetatobistriphenylphosphine mercury. To a solution of 4 grams (0.0075 moles) of mercury (II) trichloroacetate in 40 cc. of dry tetrahydrofuran (distilled from  $\text{LiAlH}_4$ ) were added 1.96 grams (0.0075 moles) of triphenylphosphine. The reaction mixture was stirred and cooled to prevent product decomposition. The solvent was evaporated and gave 4.5 grams (60.4 %) of colorless liquid (this compound decomposes on standing 1-2 days).

ANAL: Calcd. for  $\text{C}_{40}\text{H}_{30}\text{Cl}_6\text{O}_4\text{HgP}_2$

%C, 52.88 %H, 3.22 %Cl, 7.70 %P, 6.83

Found: %C, 52.15 %H, 3.88 %Cl, 8.75 %P, 5.00

10. Synthesis of Ditrifluoroacetatobistriphenylphosphine-u-diacetato-dimercury. To a solution of 1.7 grams (0.00526 moles) of mercury (II) trifluoroacetate in 50 cc. of absolute ether was added 5 grams (0.00526 moles) of diacetatobistriphenylphosphine-u-diacetatodimercury. The reaction mixture was stirred and cooled to prevent product decomposition. The precipitated colorless solid was collected in 2.5 gram (65.1 %) yield.

## SUMMARY

Objectives     The objectives of this report are:

1. The preparation and evaluation of the mercurial (II) acetates and the triphenyl phosphine complexes of mercurial (II) acetates as candidate additives for anti-oxidation, extreme pressure, and anti-wear in new lubricants. The synthetic results for the preparation of mercurial (II) acetates are tabulated in Table I.

The synthetic results for the preparation of the triphenylphosphine complexes of mercurial (II) acetates are tabulated in Table II.

Both series of compounds listed are ready for evaluation as candidate additives.

# REFERENCES

1. R. C. Evans, Frederick G. Mann, H. S. Peiser and Donald Purdie,  
J. Chem. Soc. 1209 (1940).
2. F. G. Mann and Donald Purdie, J. Chem. Soc. 1230 (1940).



TABLE I

## PREPARATION OF MERCURIAL (II) ACETATES

<u>No.</u>	<u>Compound</u>	<u>Structure</u>	<u>Physical State</u>	<u>% Yield</u>	<u>M.P.</u>
1.	Mercury (II) acetate	$(\text{CH}_3\text{COO})_2\text{Hg}$	white solid	----	dec.
2.	Mercury (II) propionate	$(\text{CH}_3\text{CH}_2\text{COO})_2\text{Hg}$	white solid	76.5	111.5-112.5
3.	Mercury (II) butyrate	$(\text{CH}_3\text{CH}_2\text{CH}_2\text{COO})_2\text{Hg}$	white solid	29.1	95-96
4.	Mercury (II) chloroacetate	$(\text{ClCH}_2\text{COO})_2\text{Hg}$	white solid	37.7	114-115
5.	Mercury (II) perfluorobutyrate	$(\text{CF}_3\text{CF}_2\text{CF}_2\text{COO})_2\text{Hg}$	pale brown liq.	30.7	----
6.	Mercury (II) benzoate	$(\text{C}_6\text{H}_5\text{COO})_2\text{Hg}$	white solid	93.6	110-112
7.	Mercury (II) trifluoroacetate	$(\text{CF}_3\text{COO})_2\text{Hg}$	white solid	94.3	122-125
8.	Mercury (II) trichloroacetate	$(\text{CCl}_3\text{COO})_2\text{Hg}$	white solid	68.8	127 dec.
9.	Mercury (II) hydroxyacetate	$(\text{HOCH}_2\text{COO})_2\text{Hg}$	white solid	40.8	114-115
10.	Mercury (II) acetamide	$(\text{CH}_3\text{CONH})_2\text{Hg}$	white solid	70.0	195

\*---Commercial preparation.

TABLE II

## PRODUCTS OBTAINED FROM TRIPHENYLPHOSPHINE AND VARIOUS MERCURY (II) ACETATES

Reactant with Triphenyl- Phosphine	Product	Structure	Yield	M.P.	Analysis					
					Carbon, %		Hydrogen, %		Phosphorus, %	
No.					Calcd.	Found	Calcd.	Found	Calcd.	Found
1.	$(\text{CH}_3\text{COO})_2\text{Hg}$ Diacetatobistri- phenylphosphine -u-diacetatodi- mercury		82.5	dec.	45.51	45.62	3.13	3.91	5.16	4.57
2.	$(\text{CH}_3\text{CH}_2\text{COO})_2\text{Hg}$ Dipropionato- bistriphenyl- phosphine-u- dipropionato- dimercury		47.0	263- 264.5	47.31	50.82	4.38	4.52	5.08	6.60
3.	$(\text{CH}_3\text{CH}_2\text{CH}_2\text{COO})_2\text{Hg}$ Dibutyrate- bistriphenyl- phosphine-u- dibutyrate- dimercury		42.3	151-2	48.99	49.21	4.58	4.96		
4.	$(\text{CF}_3\text{CF}_2\text{COO})_2\text{Hg}$ Diperfluoro- acetatobistri- phenylphosphine- mercury		63.2	220-1	45.01	45.60	2.54	2.83	5.38	6.44

TABLE II (cont'd)

Reactant with Triphenyl- No. phosphine	Product	Structure	% Yield	M.P.	Carbon, % Calcd.	Hydrogen, % Calcd.	Phosphorus, % Calcd.	Found
5. (HOCH <sub>2</sub> COO) <sub>2</sub> Hg	Dihydroxyacetato- bistriphenyl- phosphine mercury		71.8	195	54.99	54.08	9.91	4.23 7.10 6.78
6. (ClCH <sub>2</sub> COO) <sub>2</sub> Hg	Dichloroacetato- bistriphenyl- phosphine mercury		58.2	192 dec.	52.61	50.82	3.76	3.64 6.80 5.85 <sup>b</sup>
7. (C <sub>6</sub> H <sub>5</sub> COO) <sub>2</sub> Hg	Dibenzoyato- bistriphenyl- phosphine mercury		64.1	204- 205	62.05	62.20	4.16	4.64 6.41 6.37
8. (CF <sub>3</sub> COO) <sub>2</sub> Hg 3 2	Ditrifluoroacetato- bistriphenylphosphine mercury		61.6	230- 231	50.51	50.58	3.16	3.28 6.52 7.57
9. (CCl <sub>3</sub> COO) <sub>2</sub> Hg	Ditrichloro- bistriphenyl- phosphine mercury		60.4	119.	52.38	52.15	3.22	3.88

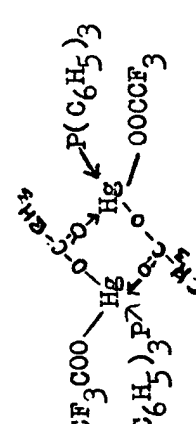
TABLE (II) (cont'd)

Reactant with Triphenyl- No. Phosphine	Product	Structure	% Yield	M.P.	Carbon, Calcd.	Hydrogen, Calcd.	Phosphorous, Calcd.	Found
5. (HOCH <sub>2</sub> COO) <sub>2</sub> Hg	Dihydroxyacetato- bistriphenyl- phosphine mercury	(C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> P Hg (C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> P	71.8	195	54.99	54.08	3.91	4.23 7.10 6.78
6. (ClCH <sub>2</sub> COO) <sub>2</sub> Hg	Dichloroacetato- bistriphenyl- phosphine mercury	(C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> P Hg (C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> P	58.2	192 dec.	52.61	50.82	3.76	3.64 6.80 5.85 <sup>b</sup>
7. (C <sub>6</sub> H <sub>5</sub> COO) <sub>2</sub> Hg	Dibenzoato- bistriphenyl- phosphine mercury	(C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> P Hg (C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> P	64.1	204- 205	62.05	62.20	4.16	4.64 6.41 6.37
8. (CF <sub>3</sub> COO) <sub>2</sub> Hg	Ditrifluoroacetato- bistriphenylphos- phine mercury	(C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> P Hg (C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> P	61.6	230- 231	50.51	50.58	3.16	3.28 6.52 7.57
9. (CCl <sub>3</sub> COO) <sub>2</sub> Hg	Ditrichloro- bistriphenyl- phosphine mercury	(C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> P Hg (C <sub>6</sub> H <sub>5</sub> ) <sub>3</sub> P	60.4	119.	52.38	52.15	3.22	3.88

<sup>a</sup> Oxygen present: Calcd. 11.02; Found, 11.14.  
<sup>b</sup> Chlorine present: Calcd. 7.79; Found, 8.21.

TABLE II A

PREPARATION OF DITRIFLUOROACETATOBISTRIPHENYLPHOSPHINE

No.	Reactant with Diacetato- bistriphenylphosphine -u-diacetatodimercury	Product	Structure	% Yield	M.P.	Carbon, %		Hydrogen, %	
						Calcd.	Found	Calcd.	Found
1.	$(CF_3COO)_2Hg$	Ditrifluorencetato bistriphenylphosphine -u-diacetato-dimercury		65.1	189- 190	41.60	41.87	2.84	2.92